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Characterization of synthetic pargasitic amphiboles

( $\text{NaCa}_2\text{Mg}_4\text{M}^{3+}\text{Si}_6\text{Al}_2\text{O}_{22}(\text{OH},\text{F})_2$ ;  $\text{M}^{3+} = \text{Al, Cr, Ga, Sc, In}$ ) by infrared spectroscopy, Rietveld structure refinement, and  $^{27}\text{Al}$ ,  $^{29}\text{Si}$ , and  $^{19}\text{F}$  MAS NMR spectroscopy

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For deposit: Table 3

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Table 3: Run product descriptions.

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Pargasite:  $M^{3+}=Al$ . Pargasite was synthesized readily with yields of 80 to 95 percent. Best results were obtained at 800° to 900°C, 2 to 3 kbar water pressure. Pargasite formed clear, colorless prisms up to 40 microns long and 8 microns wide. Run length had no noticeable effect on grain size and amphibole yield; runs of 27 and 1126 hr had similar yields, grain size and infrared spectra (see below). Cell dimensions of pargasites are consistent with those of previous workers.

Chromium-pargasite:  $M^{3+}=Cr$ . Substitution of Cr for octahedral Al reduced amphibole yields to about 80 percent. Chromium-pargasite formed pale green, slightly pleochroic prisms up to 24 microns long and 1 to 2 microns wide. Overall grain size was finer than pargasite, and crystals tended to be more acicular in habit. Abundant eskolaite ( $Cr_2O_3$ ) in all runs suggests that the amphibole was not on composition; the amounts of Cr in clinopyroxene and spinel are uncertain. Cell dimensions (Table 2) of three typical runs are similar with cell volumes 4 to 6 Å<sup>3</sup> larger than those of pargasites. The increase in volume is due almost entirely to an increase in b, suggesting that substantial amounts of Cr had replaced Al in the octahedral strip.

Gallium-pargasite:  $M^{3+}=Ga$ . Poor amphibole yields (20 to 30 percent) were obtained at 1 kbar and temperatures less than 800°C; the run product was mostly clinopyroxene with minor plagioclase, forsterite, nepheline and three different layer silicates with approximate basal spacings of 14.8, 12.2 and 9.8 Å. Raising either the pressure to 2 kbar or the temperature above 800°C, increased amphibole yields to more than 90 percent. Amphibole formed clear, colorless crystals up to 15 microns long and 2 to 4 microns wide. Layer silicates were not present. The amphibole grown at 758°C, 1 kbar, has cell dimensions (GaPA-A1, Table 2) that are edenitic in character, rather than pargasitic (lower a,

$\beta$ , higher  $b$ ). Apparently little, if any Ga was incorporated at the octahedral sites. At 817°C, 2.1 kbar, the cell dimensions of the amphibole are very similar to those of chromium-pargasites (Table 2). Because the ionic radii of Cr (0.615 Å) and Ga (0.620 Å) in octahedral coordination are almost identical, this similarity probably reflects the same degree of  $M^{3+}$  substitution for Al in these amphiboles. Gallium not in amphibole probably in the silicate phase or, where present, in layer silicates.

Scandium-pargasite:  $M^{3+}=Sc$ . Scandium-pargasite was synthesized but yields were never more than about 90 percent. Amphibole formed clear, colorless crystals up to 11 microns long and 1 to 2 microns wide.  $Sc_2O_3$  was a minor phase in all run products, indicating that the amphibole was off-composition. Cell dimensions (Table 2) show that the amount of Sc at octahedral sites is high; the cell volume of scandium-pargasite is about 15 Å<sup>3</sup> larger than that of pargasite.

Indium-pargasite:  $M^{3+}=In$ . About 90 percent yields of amphibole were obtained in 2 kbar runs. Amphibole formed pale yellow, faintly pleochroic crystals up to 8 microns long and 1 micron wide. Although the cell volume of this amphibole (Table 2) is about 9 Å<sup>3</sup> larger than that of pargasite, it is 6 Å<sup>3</sup> smaller than that of scandium-pargasite, indicating only partial substitution of In for Al in the octahedral strip. Furthermore, all run products contained unreacted  $In_2O_3$ .

Fluor-pargasite:  $M^{3+}=Al$ . All runs on the fluor-pargasite mix prepared with Corning silica glass as  $SiO_2$  source yielded more than 90 percent amphibole. Mixes prepared with  $H_2SiO_3 \cdot nH_2O$  failed to grow amphibole. Fluor-pargasite formed clear, colorless crystals up to 16 microns long and 4 microns wide in isothermal runs. Non-isothermal runs produced large crystals between 0.02 and 1 mm in length. In both types of experiments, products were similar except for minor fluorite in non-isothermal experiments. Cell dimensions of amphiboles

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with either cooling history are similar (Table 2).

Chromium-fluor-pargasite:  $M^{3+}=Cr$ . As in chromium-pargasite, Cr substitution reduced yields to about 80 percent in isothermal runs. Little amphibole was formed in the single non-isothermal run. Chromium-fluor-pargasite formed pale green, slightly pleochroic, blocky to prismatic crystals up to 18 microns long and 6 microns wide. Cell volumes are 3 to 4  $\text{\AA}^3$  larger than that of fluor-pargasite. This increase is due mainly to an increase in  $b$ , suggesting Cr occupancy in the octahedral strip.

Gallium-fluor-pargasite:  $M^{3+}=Ga$ . Gallium-fluor-pargasite formed about 85 to 90 percent blocky to prismatic, clear, colorless crystals up to 14 microns long and 5 microns wide in isothermal experiments. Larger crystals to 0.5 mm long were formed in non-isothermal runs. Cell dimensions were not affected by run history and are similar in both runs. The cell dimensions of an isothermal run analysed by the Rietveld method are systematically larger than the other two; it may have had more Ga substituted for Al than the other two.

Scandium-fluor-pargasite:  $M^{3+}=Sc$ . Scandium-fluor-pargasite formed about 85 to 95 percent of the products and occurred as blocky to prismatic, grains up to 23 microns long and 5 to 16 microns wide. The single non-isothermal run grew crystals up to 0.5 mm in length but yielded only about 75 percent amphibole. The cell volume is about 20  $\text{\AA}^3$  larger than that of fluor-pargasite which suggests that most of the octahedral Al was replaced by Sc. Note that the volume of scandium-fluor-pargasite is larger by about 2  $\text{\AA}^3$  than that of scandium-pargasite. As the volume of hydroxy-amphibole should be larger than that of its fluorine analogue, it is probable that the scandium-pargasite has a lower Sc occupancy than the scandium-fluor-pargasite.

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